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TECHNICAL MEMORANDUM

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EFFECTS OF SOLVENT CONTAMINATION
ON
BALLISTIC PERFORMANCE
OF
M5 FLAKE PROPELLANT
IN
SMALL EJECTION TYPE ITEMS

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SECTION I

INTRODUCTION

Low ejection velocities have been encountered in a number of devices using the M5 Flake Propellant. There was some evidence that the difficulty might be due to solvent contamination of the propellant. The purpose of this study was to determine by laboratory techniques the effects of M5 propellant exposure to solvent laden atmospheres ... using the solvents which might be present in the assembly. If this showed in fact that this was a potential problem, investigation of methods of revitalizing the propellant by solvent removal would be studied to permit reclamation of reject lots.

SECTION II

SUMMARY

The effects of solvent contamination of propellant charges used in "jump up" devices was studied. It was found that any solvent contamination will lower the quickness of M5 flake propellant. Five percent of solvent will lower performance by 30% or more, depending on the solvent. Recovery of original propellant ballistic properties is possible if the amount of solvent absorbed has not reached the point where a change in the physical form of the propellant can occur. The presence of foreign materials, such as graphite and condensed water in the pressure chamber, has little effect on the ballistic performance of the propellant..

SECTION III

CONCLUSIONS

1. Propellant cups, removed from "jump up" assemblies from lots in which malfunction had occurred, contained sufficient solvent vapors to reduce propellant quickness to a point which could cause malfunctions.

2. The functioning of M5 flake propellant is extremely sensitive to solvent contamination. The presence of 5% of solvent will cause a reduction of at least 30% in quickness.

3. Propellant in "sealed" cups will readily absorb large amounts of solvent vapors. Removal of solvent by heating will not fully restore ballistic properties when large amounts of solvents (which soften nitro-cellulose) are absorbed.

4. Revitalization of "jump up" assemblies is possible by heating at 130⁰ F for 40 hours if solvent absorption has not reached the point at which physical distortion of the grains can occur.

5. The presence of graphite and water should have little effect on the ballistic performance of the M5 flake propellant.

6. Absorption of solvent causes contraction in the thickness of crimped cups. Therefore, there should be no interference with moving parts.

7. Absorption of solvents tested caused swelling of gaskets.

SECTION IV

RECOMMENDATIONS

1. Eliminate the use of solvents wherever possible in the assembly of ejection type items.
2. Where use of solvents cannot be avoided, parts must be dried thoroughly before assembly of units.
3. Reconditioning of assemblies containing solvent exposed propellant is possible if the solvent content has not gone too high. Heating of the assembly at 130^oF for 40 hours with venting to the atmosphere should restore proper functioning.

SECTION V

STUDY

Background and Analysis

Several "jump up" devices have been developed which use small quantities of M5 flake (about .0025 inch thick by 0.30 inch diameter) propellant. The combination of high burning rate and large surface area of the propellant produces a very quick burning charge which produces the rapid pressure pulse needed. Recently, low ejection velocities or no ejection has been encountered in some of these items although the propellant was consumed. Solvent contamination was suspect because (1) contamination by solvent vapors would be expected to reduce the quickness of the propellant and result in a reduced impulse and (2) several possible sources of solvent vapors (namely cements and lacquer) are present in the item.

Examination of the items showed that under normal assembly conditions it is entirely possible to seal materials high in solvent content (cements, lacquers, etc.) into the item. This would then permit the propellant charge (in crimped aluminum cups, sometimes lacquer sealed) to remain exposed to solvent vapors for long periods of time. Some of these vapors are good nitrocellulose solvents and might be expected to be readily absorbed. Others, like toluene for example, while not solvents for nitrocellulose, are absorbed on the large surface area presented by the finely divided propellant.

In one instance, items containing M5 propellant were assembled in proximity to bagged propellant charges. Residual solvents in M1 propellant

are alcohol and water. Possible contamination from this source was made part of this study.

Some peripheral investigations were undertaken in addition to solvent effects on the propellant. Graphite is sometimes used to lubricate arming mechanisms and condensed moisture could be present in the item; the effects of the presence of these materials were checked. Propellant swells when solvent is absorbed; its effects on dimensions of the loaded crimped cups was determined. Expansion might effect moving parts. Rubber gaskets are used on some assemblies and the effect of their exposure to solvent atmospheres was determined.

PROGRAM

The program undertaken was designed to establish: (1) whether absorption of solvent vapors had occurred in crimped propellant cups from lots in which malfunctions had occurred, and if so what its effect was on the propellant performance; (2) what are the effects of contamination by different solvents on the ballistic performance of M5 flake propellant; (3) to what extent will solvent absorption occur in "sealed" (crimped and in some instances also lacquered) aluminum cups containing the propellant; (4) whether revitalization of the assembled unit is possible by removal of solvent by heating at 130⁰ F for 40 hours; (5) miscellaneous effects as presence of graphite and water in the firing chamber, dimensional changes in crimped propellant cups when exposed to solvent vapors and effects of solvent vapors on rubber gaskets used in some assemblies.

1. Examination of Propellant Involved in Malfunctions

To determine whether erratic functioning of complete assemblies could have resulted from solvent contamination of the propellant, cups were removed from lots where malfunction had occurred. These units had been loaded with M5 propellant HES 5250.92, Lot 3. The propellant was removed and fired in the closed bomb for comparison with a sample of the original propellant. Samples also were sent to the laboratory for total volatiles analysis. These results are in Table I. This data is considered to be somewhat low with respect to volatiles content since samples were shipped from the West Coast without taking any precautions to prevent solvent loss and the bottles in which the propellant was stored after removal and transfer to the laboratory were found to be not vapor tight. All these samples had the characteristic odor of butyl or ethyl acetate. Volatiles content was well above that obtained for the original powder lot.

All these samples showed a loss of quickness of 25-34% when compared with the original propellant. This quickness is in line with a solvent content of from 3 to 5% as established in subsequent tests.

2. Solvent Absorption in Propellant

The effects of solvent contamination on loose M5 flake propellant was determined by exposing samples to saturated solvent vapors in closed containers at room temperature. These samples were exposed for varying lengths of time and the percent of solvent absorption determined

from gain in weight. The solvent-contaminated samples were then tested in the closed bomb for relative quickness and force ... compared to the uncontaminated propellant as the standard.

Table II gives this data. Figure I is a plot of quickness vs. percent absorbed solvent for each type of solvent used. The quickness falls off rapidly with percent solvent for all the solvents tested.

In Table II, note that the propellant exposed to residual solvent vapors from M1 propellant for 47 days showed no absorption and little change in quickness.

When large amounts of nitrocellulose solvents (like ethyl and butyl acetates) were absorbed, the propellant grains fused and caked. On continued exposure some samples absorbed as much as 150% of their original weight. The rate of solvent absorption appears related to the vapor pressure of the solvent. The total amount of solvent absorbed is a function of its solvent power for the M5 propellant. Table VII and Figure 2 give the properties of solvents frequently found in lacquers and cements.

To determine whether removal of the solvent vapors would restore propellant ballistic properties, samples were exposed to solvent vapors and then redried in an oven at 130^o F until all the absorbed vapors had been removed. Table III shows the results with samples exposed to ethyl acetate and re-dried. Re-drying restored original ballistic properties to the propellant. Actually, continued drying gave a slight

weight loss to below original weight of the sample, indicating a loss of volatiles, which increased the quickness to above its original value. The last four items in Table III show that drying at 130^o F can raise the quickness to as much as 13% above the original value. Re-exposure of oven dried samples to the atmosphere permitted re-absorption of moisture which restored quickness to its original value (Test No. 3 in Table III).

When solvent absorption exceeded 30%, samples fused and caked so badly that tests were discontinued. Caking of the propellant began after 6% of ethyl and butyl acetates were absorbed.

3. Tests of Sealed & Crimped Cups

To test solvent absorption of propellant in aluminum cups which had been crimped and lacquered, a series of these cups were exposed to saturated solvent atmospheres at room temperature and weighed at intervals. After the time periods specified in Table IV, the cups were weighed and the solvent absorption calculated. The results obtained were similar to those obtained for loose propellant except that the rates were naturally much lower. The seal on these cups is obviously quite pervious to solvent vapors. The cups now laden with solvent vapors were then placed in an oven at 130^o F and dried to constant weight. The propellant was removed from the cups and tested in the closed bomb. The quickness of the samples exposed to ethyl and butyl acetates was not completely recovered (Table IV).

There was speculation whether solvent absorption might cause changes in dimensions of the cups. An increase in thickness might cause interference with the arming device in some of these items. Since swelling of the bulk propellant occurred when large amounts of ethyl or butyl acetate were absorbed, this might cause an expansion in thickness of these thin walled containers. Testing showed that this had not occurred. On the contrary, a decrease in thickness had actually taken place after solvent absorption, which was not recovered after drying. The average change in thickness of cups is included in Table IV. Since in the loaded cups the propellant is under slight compression, apparently softening of the propellant by the solvent permitted the flakes of propellant to be pressed together. This reduction in surface and subsequent loss in quickness could explain why propellant in cups which had been allowed to absorb large amounts of ethyl and butyl acetates did not completely recover in quickness.

4. Revitalization of Complete Assemblies

Experiments to determine conditions for revitalization of complete assemblies showed that heating of the unit at 130^o F for 40 hours (with venting) produced satisfactory functioning. A large number of lots were made acceptable by this technique although occasional lots were found which did not respond. This may be because the solvent absorption had gone too far or because malfunction was the result of other causes.

5. Miscellaneous Tests

To check the effects of the presence of excess graphite in the chamber, varying amounts of graphite (up to 85 mg) were injected into some of the revitalized units before functioning tests were run. The only failure among these was one which showed a residual solvent odor after firing (Table V) and the failure was attributed to solvent contamination. To further check the effect of graphite on the quickness of the propellant, tests of M5 flake propellant also were run in the closed bomb with 2% of graphite in the chamber, but not mixed in with the propellant. Table VI shows that a slight reduction in quickness does occur, probably because of absorption of heat by the graphite. However, the loss in quickness is not significant in terms of functioning of the unit.

The effects of water not absorbed by the propellant also were determined in the closed bomb and found to result in a slight but not significant loss in quickness of the propellant.

The complete assemblies contained sealing gaskets made of MIL-R-3065B SB715 BE, E_3F_2 rubber. The effects of solvent vapors on the dimensions of the gaskets was investigated. These gaskets were exposed to saturated vapors of ethyl acetate, butyl acetate, toluene and cyclohexanone and weighed and measured after overnight exposure. Then they were air dried until most of absorbed solvent vapors were dissipated and dried in an oven overnight at 130°F. The weights and dimensions were checked again. Table VIII shows the data obtained.

Large amounts of solvent vapors were absorbed in every case and a great deal of swelling occurred. The gaskets resumed their original dimensions after drying.

APPENDIX A

TABLES

TABLE I

Comparison of Propellant Taken From Defective Lots of Loaded Cups
Compared with Original Propellant as Standard of 100%

<u>Lot No.</u>	<u>Avg. Relative Quickness, %</u>	<u>Relative Force, %</u>
Std 5250.92 Lot 3	100.0	100.0
Prop. From JA-SR-77A	74.6	98.2
Prop. From JA-SR-77A1	74.9	101.3
Prop. From JA-SR-77B1	67.9	98.2
Prop. From JA-SR-77B2	66.0	95.1

Total Volatiles*

Std 5250.92 Lot 3	.7**
SR-77A	2.48
SR-77A1	1.38
SR-77B1	1.88
SR-77B2	1.51

*These values are considered low because of loss of vapors during
handling of the samples prior to testing.

** Approximate value

TABLE II

Effects of Exposure to Solvent Vapors on Ballistic Properties
of M5 Flake Propellant Lot HES 5250.89 Lot 2

Ethyl Acetate			
<u>Solvent Absorbed, % of Original Weight</u>	<u>Time of Exposure, Hours</u>	<u>Ave. Rel.* Quickness</u>	<u>Rel.* Force</u>
1.08	1/2	80.8	100.4
1.29	1 1/2	78.4	99.3
3.31	3	67.7	99.3
5.95	5	50.8	100.4
25.7	17	18.9	89.5
<u>Butyl Acetate</u>			
.14	1/2	97.1	100.5
.11	1	97.9	100.0
.74	2	88.8	100.5
2.84	4	82.2	102.1
15.10	18	39.7	95.9
<u>Toluene Vapors</u>			
.10	1	95.4	99.8
.32	3	96.9	100.3
.51	6	95.5	98.7
2.77	20	75.7	100.5
4.81	68	63.7	102.3

TABLE II (Cont'd)

Cyclohexanone Vapors

<u>Solvent Absorbed, % of Original Weight</u>	<u>Time of Exposure, Hours</u>	<u>Avg. Rel* Quickness</u>	<u>Rel.* Force</u>
0	1/2	98.9	100.0
0	1	100.4	100.0
-.11	2	100.4	100.5
.02	4	95.1	100.5
1.03	18	54.3	96.4
30.4	95	15.2	83.1
<u>Water Vapor</u>			
.34	2	96.0	98.4
.51	4 1/2	94.1	100.5
.61	6	91.0	100.0
1.10	16	87.3	100.5
15.7	60 days	39.5	98.4
<u>M1 Propellant Vapors</u>			
-.03	47 days	97.6	100.5

*Compared to original Sample as standard of 100%

TABLE III

Effects of Exposure to Ethyl Acetate Vapors and Redrying on
Ballistic Properties of M5 Flake Propellant

<u>Lot HES 5250.89 Lot 2</u>					
<u>Solvent Absorbed, % Of Original Weight</u>	<u>Time of Exposure, Hours</u>	<u>Redrying Time at 130°F, Hrs.</u>	<u>Final Weight, % Change</u>	<u>Avg. Rel ^a Quickness, %</u>	<u>Relative ^a Force, %</u>
0.3	1/2	1 1/2	-0.6	107.1	99.0
1.8	1 1/2	2 3/4	-0.4	108.8	100.5
6.1	2 2/3	2 1/2 <u>c</u>	0.4	99.5	100.0
23.5	16	4	1.4	95.3	99.7
23.6	16	4	1.5	93.9	100.3
29.7	17	21 1/2	-0.7	107.5	99.7
30.6	17	21 1/2	-0.8	106.5	99.7
0.0 <u>d</u>	none	1 1/2	-0.7	110.6	99.0
0.0 <u>d</u>	none	2 1/2	-0.8	113.3	100.0
0.0 <u>d</u>	none	4	-0.4	107.8 <u>b</u>	99.7 <u>b</u>
0.0 <u>d</u>	none	21 1/2	-1.0	111.7 <u>b</u>	99.7 <u>b</u>

a Compared to original sample as standard of 100%

b Average of 2 samples

c Exposed to atmosphere 16 hours after drying

d Blank

TABLE IV

Functioning of Blu/4 Propellant Cups After Exposure to Solvents and Redrying

Solvent Exposure Days	% Absorbed Solvent	Change in Thickness Inch	Drying Time Days at 130°F	Final Weight of Charge, % of Orig. Wt.	Relative ^a Quickness	Relative ^a Force
Blank - none	none	none	none	none	103.1	99.9
Water Vapor - 17 days	5.3%	-.003	7 days	-.1%	100.7	101.6
Oven Dried - none	none	-.002	11 days	-0.3%	102.4	101.6
Ethyl Acetate - 5 days	49.9%	-.009	7 days	-0.1%	66.1	97.3
Butyl Acetate - 17 days	43.2%	-.004	7 days	-0.3%	24.8	82.5
Butyl Acetate - 17 days	43.2%	-.004	14 days	-0.9%	67.4	93.2

^a Compared to HES 5250.92 Lot 4 which was used to load these rounds.

TABLE V

Functioning of Assemblies after Addition of Graphite

<u>Lot No.</u>	<u>Date</u>	<u>No. Tested</u>	<u>Graphite Added</u>	<u>Function</u>
72-6	2 Feb	4	15 mg	3 OK, 1 failed Heavy solvent odor in case after firing in failed unit.
72-5	6 Feb	1	85 mg	1, OK
		10	none	10, OK
		4	20 mg	4, OK
		1	85 mg	1, OK
72-6	6 Feb	10	none	10, OK
		15	none	15, OK
72-8	6 Feb	4	20 mg	4, OK
		1	85 mg	1, OK
		10	none	10, OK

TABLE VI

Closed Bomb Test of Lot HES 5250.89 Lot 2
With Graphite and Water Added

<u>Condition</u>	<u>Avg. Relative Quickness</u>	<u>Avg. Relative Force</u>
Graphite - .3g (2% of sample wt.) in front of sample	94.9	98.7
Graphite - .3g (2% of sample wt.) in back of sample	97.5	98.2
Water - 5 drops (2% of sample wt.) in front of sample	97.6	99.2

TABLE VII

Properties of Common Solvents Found in Lacquers and Cements

<u>Solvent</u>	<u>Formula</u>	<u>Formula Wt.</u>	<u>Boiling Pt. at 760 mm Hg</u>	<u>Solvent for NC</u>
Acetone	$\text{CH}_3 \text{ CO } \text{CH}_3$	58	56.15°C	Yes
Butyl Acetate	n- $\text{CH}_3 \text{ CO}_2 (\text{CH}_2)_2 \text{C}_2 \text{H}_5$ iso- $\text{CH}_3 \text{ CO}_2 \text{CH}_2 \text{CH}(\text{CH}_3)_2$	116	n-125.0 iso-118.0	Yes
Cyclohexane	$\text{CH}_2 (\text{CH}_2 \text{CH}_2)_2 \text{CH}_2$	84	80.7	No
Cyclohexanone	$\text{CH}_2 (\text{CH}_2 \text{CH}_2)_2 \text{CO}$	98	156	Partially
Ethyl Acetate	$\text{CH}_3 \text{ CO}_2 \text{C}_2 \text{H}_5$	88	77.1	Yes
Ethyl Alcohol	$\text{CH}_3 \text{CH}_2 \text{OH}$	46	78.4	No
Ether (diethyl)	$(\text{CH}_3 \text{CH}_2)_2 \text{O}$	74	34.6	No
Toluene	$\text{C}_6 \text{H}_5 \text{CH}_3$	92	110.6	No
Xylene	$\text{C}_6 \text{H}_4 (\text{CH}_3)_2$	106	138 to 144	No
Water	$\text{H}_2 \text{O}$	18	100	No

- Hydrocarbons and alcohols are generally non-solvents for nitrocellulose
- Esters and ketones are the best solvents
- Straight chain compounds are better solvents than cyclic compounds
- Some non-solvents make good co-solvents, for example: ether and ethanol. (SEE above)
- Some non-solvents can affect fibre physical properties (induce swelling) if permitted to diffuse into matrix.

TABLE VIII

Effect of Solvent Vapors on MIL-R-3065B,
SB715BE, E_3F_2 Rubber Gaskets

<u>Solvent</u>	<u>Gain in Wt. % of original</u>	<u>Condition after 18 hrs. exposure</u>	<u>Condition after redrying</u>
Ethyl Acetate	40	Swelled, O.D. Increased 7/16"	Returned to original dimensions after air drying
Butyl Acetate	53	Swelled, O.D. Increased 7/16"	Returned to original dimensions after air drying
Toluene	81	Swelled, O.D. Increased 3/4"	Returned to original dimensions after air drying
Cyclohexanone	24	Swelled, O.D. Increased 3/16"	Returned to original dimensions after oven drying

APPENDIX B

FIGURES

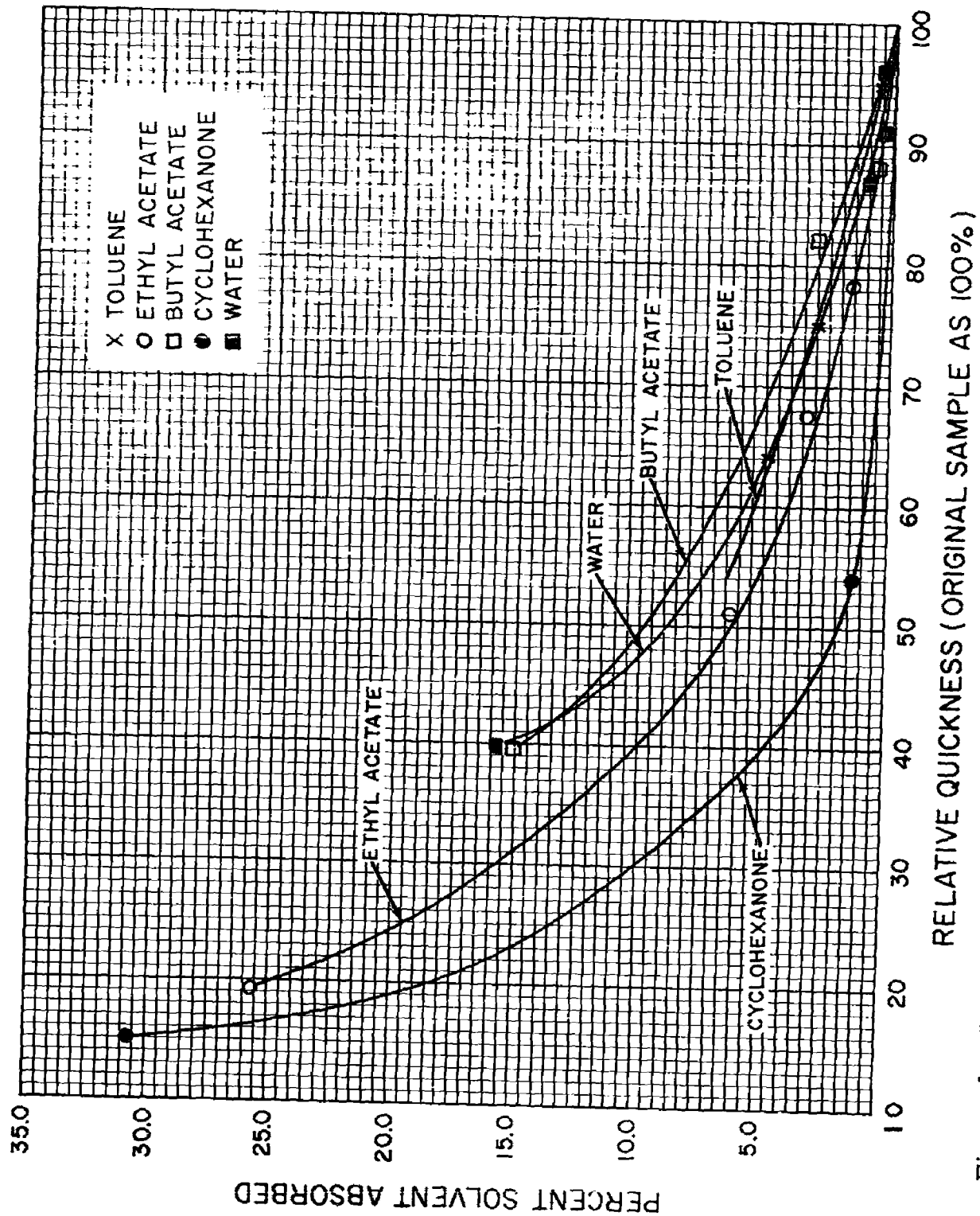


Figure 1. Loss of Quickness as a Function of Absorbed Solvent for M5 Flake Propellant

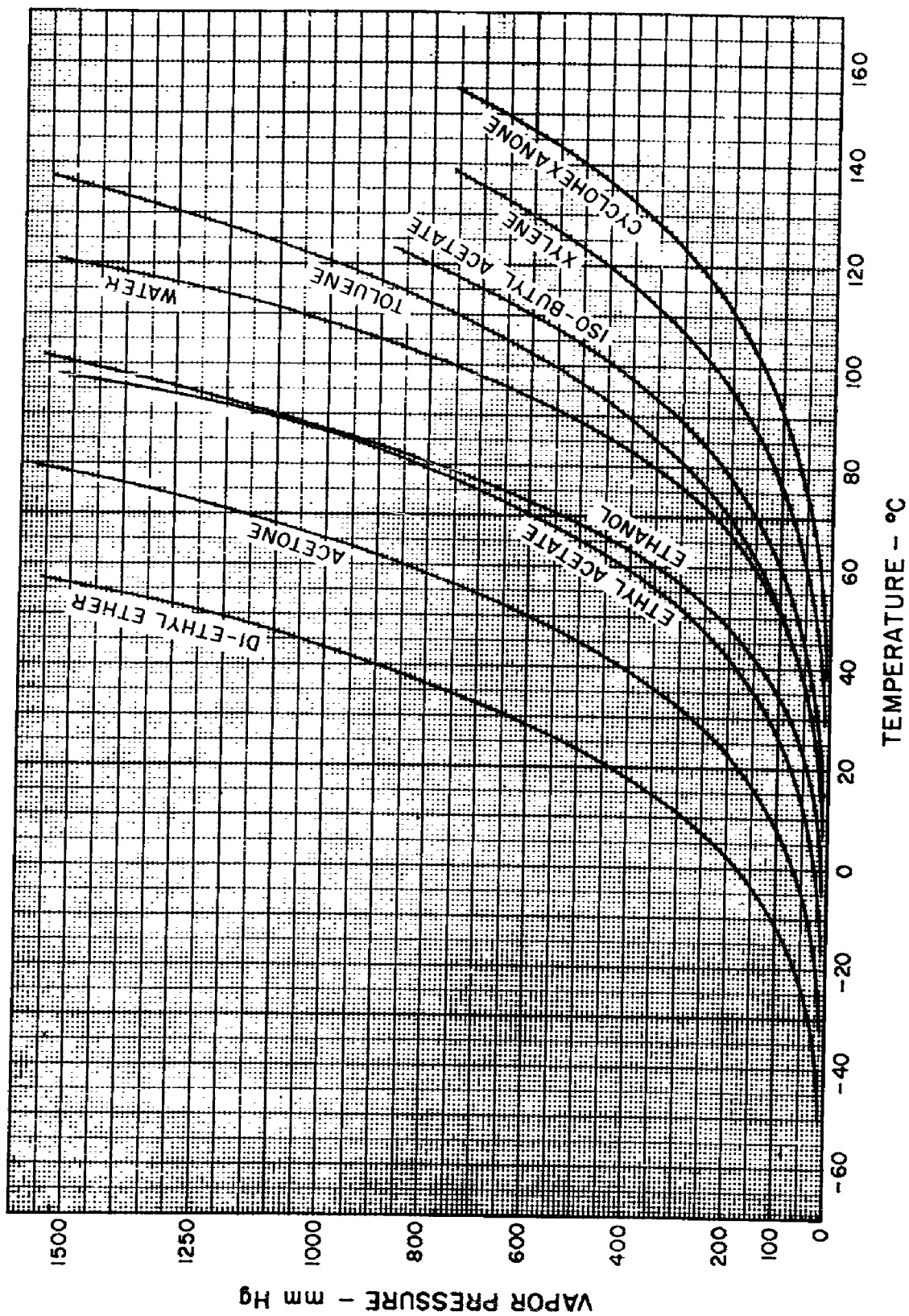


Figure 2. Vapor Pressure vs. Temperature for Common Solvents

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